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L4: Entry 4 of 71

File: PGPB

Mar 13, 2003

DOCUMENT-IDENTIFIER: US 20030047533 A1

TITLE: Method for removing a sacrificial material with a compressed fluid

Detail Description Paragraph (19):

[0028] The sacrificial layer comprises an organic material, a carbon compound, that is deposited by, for example, spray-on or spin-coating. In one embodiment, the organic material is mixed with a solvent and deposited on a substrate. The solvent is preferably any known solvent for dissolving the organic material to be used, such as a supercritical fluid and/or a volatile organic solvent. The solvent is selected based on good handling, spinning and film forming properties (for spin on non-supercritical embodiments). In a preferred embodiment, a supercritical fluid, such as carbon dioxide, along with a cosolvent, dissolves a polymer and deposits the dissolved polymer on a substrate as a sacrificial layer.

Detail Description Paragraph (28):

[0037] In the alternative to CVD, the organic sacrificial layer can be deposited with a liquified gas, such as liquid CO₂, or with supercritical CO₂. Polymers, particularly fluorinated polymers can be formed as a film on a solid substrate using dip-coating with liquid carbon dioxide as the solvent. Also, liquid carbon dioxide can be as the solvent for spin coating photoresist (e.g. fluorocarbon photoresist) on a substrate (glass, silicon, etc.). Or, supercritical CO₂ could be used instead of liquid CO₂ in a RESS (Rapid Expansion of Supercritical Solution) to form a particulate coating as the sacrificial layer.

Detail Description Paragraph (29):

[0038] In order to use liquid CO₂ as a solvent for spin coating, a high-pressure coating chamber with rotating chuck is useful. To spin coat from liquid CO₂, the resist should be soluble in liquid CO₂, or soluble in whatever supercritical fluid is used (alone or with a cosolvent). An example of liquid CO₂ as a medium for spin coating is disclosed in Kendall et al. "Liquid Carbon Dioxide Spin Coating Process for Deep-UV Photoresists", conference paper from Fluorine in Coatings III, Orlando, Fla. Jan. 25-27, 1999, paper 34; and Hoggan et al. "Spin Coating and Photolithography using Liquid and Supercritical Carbon Dioxide", conference paper from ACS, New Orleans, La. Aug. 22-26, 1999, pp 47-8, both being incorporated herein by reference. Without a cosolvent, fluorinated polymers are preferred for their solubility in liquid CO₂. For example, a fluorinated octyl methacrylate copolymer can be dissolved in liquid CO₂ and spin coated in a high-pressure spin coating apparatus. And, if a photoacid generator is used in conjunction with the fluorinated polymer, the photoacid generator can be fluorinated to improve solubility in the liquid CO₂ (e.g. 2-perfluorohexyl-6-nitrobenzyl tosylate).

Detail Description Paragraph (30):

[0039] Instead of liquid CO₂, supercritical CO₂ can be used in a RESS process. In RESS, a product in, for example a supercritical carbon dioxide solution, which product is provided for the RESS process or is chemically formed upstream, is deposited as micron sized particles by rapid expansion through a nozzle. The solution is preferably heated to around 80 degrees C. before expansion and/or the pressure is reduced to 70 bar, where the product is insoluble. The expansion nozzle may be a short length of stainless steel capillary or a fine hole cut by laser in a stainless steel plate. The flow may be supersonic or subsonic in the nozzle. In the RESS process, non-volatile solutes are dissolved in a supercritical fluid, which results in a solute laden supercritical phase. A relatively small change in pressure of the supercritical phase can lead to a large decrease in solvent density, and hence solvent power. By the rapid expansion, or depressurisation, of the supercritical solution, a high supersaturation can be obtained. This high supersaturation leads to high nucleation rates and the

precipitation of very fine particles when the solution is rapidly expanded through the nozzle. The rapid expansion creates uniform conditions within the nucleating medium so that the precipitated particles have a narrow particle size distribution. Particle size and distribution can be controlled by manipulating RESS operational parameters such as the geometric characteristics of the nozzle, pre-expansion temperature and pressure, and expansion temperature and pressure, and the concentration of the solute in the supercritical solution. See, for example C. J. Chang et. al. "Precipitation of Microsize Organic Particles from Supercritical Fluids" AlChE Journal Vol.35, No 11, p1876, (1989), and D. W. Matson et.al: "Rapid Expansion of Supercritical Fluid Solutions: Solute Formation of Powders, Thin Films, and Fibers" Ind. Eng.Chem.Res, 26, p2298, (1987). A cosolvent can also be used along with supercritical carbon dioxide, such as disclosed in J. W. Tom et.al.: "Application of Supercritical Fluids in The Controlled Release of Drugs" Supercritical Fluid Engineering Science, Chapter 19, p238, (1993). Any of the wide variety of types of poly (tetrafluoroethylene), including for example Teflon AF (family of amorphous copolymers based on bistrifluoromethyl, difluoro, dioxole, and other fluorine containing monomers), could be deposited with liquid CO2 or supercritical CO2 as outlined above. Other fluorinated polymers, and other polymers with a cosolvent incorporated into the polymer backbone, could be deposited as above. Methods for coating substrates using carbon dioxide are disclosed in U.S. Pat. Nos. 6,165,559, 6,165,560, and 6,200,637, the subject matter of each being incorporated by reference, as well as in WO 027544 assigned to North Carolina State University. Prior to deposition of the fluoropolymer on the substrate (whether in an atmosphere of liquid or supercritical CO2 or by using a traditional solvent), the fluoropolymer can be synthesized in an atmosphere of supercritical CO2, such as in WO 00/68170, U.S. Pat. No. 5,981,673 or U.S. Pat. No. 5,922,833, each assigned to Univ. of N. Carolina, Chapel Hill, the subject matter of each being incorporated herein by reference.

Detail Description Paragraph (31):

[0040] Spin-on organic (or organic-inorganic hybrid) low-k materials can also be used for the sacrificial layer of the present invention. Examples include FLARE.TM. (an organic spin-on polymer for use as a low-k interlayer dielectric), HOSP.TM. (a spin-on hybrid siloxane-organic polymer), ACCUFLO.TM.T-13EL (an organic polymer in an organic solvent system) 314, 214 Spin-On Glass (SOG) series (a family of siloxane polymers), AccuGlass.TM.T-12B Spin On Glass (belongs to the methylsiloxane family of polymers) and 311, 211, 111 Spin On Glass (SOG) series (family of methylsiloxanes that combine organic groups on an inorganic polymer backbone), SiLK.TM. (a spin-on organic polymer deposited using a conventional spin-coater), Cyclotene.TM. (derived from B-staged bisbenzocyclobutene monomers), and PTFE spin-on films from W. L. Gore (Elkton, Md.) and CVD PTFE films from SVG Thermco Group. These low-k materials can be deposited by spin-on with traditional solvents (e.g. mesitylene, gamma butyrolactone) or in liquid carbon dioxide alone or with small amounts of cosolvent. The siloxane-based low-k materials are useful for being able to be deposited and removed with liquid or supercritical carbon dioxide without the need for cosolvents (or with minimal amounts of cosolvents). Many low-k materials are also beneficial in that they can be directly patterned (no photoresist for patterning)--see, for example, Weibel G L, Lewis H G P, Gleason K K, Ober C K. "Patternable low-k dielectrics developed using supercritical CO2", Polymer Preprints, 2000, 41(2), 1838-1839, incorporated herein by reference. Adhesion promoters can also be used both before and after depositing the low-k material.

Detail Description Paragraph (33):

[0042] Solvents are needed for depositing the sacrificial material on the substrate, for patterning the material if needed, and eventually removing the sacrificial material in order to release the micromechanical structures. The release, in accordance with the present invention, is in a solvent that is a supercritical fluid. As will be discussed further herein, the supercritical fluid can be selected from a wide variety of fluids that can be provided in a supercritical state. A cosolvent is not needed. Examples include, ethylene, xenon, water, toluene, carbon dioxide, nitrous oxide, methanol, n-pentane, ethane, propane, isopropanol, n-butane and ammonia. If supercritical carbon dioxide is used, and the organic material of the sacrificial layer is an organosilicate or a fluoropolymer, the supercritical CO2 can be used without a cosolvent to remove the sacrificial material to release the micromechanical structures (the supercritical CO2 without cosolvent can also be used for depositing the sacrificial layer (e.g. in a RESS process), or liquid CO2 without cosolvent can be used for sacrificial layer spin-on as disclosed above). Of course, there are many non-fluorous polymers with high solubility in supercritical CO2--see, for example, Sarbu et al. "Non-Fluorous Polymers with Very High Solubility in Supercritical CO2 Down to Low Pressures", Nature, vol 405, no.6783, 2000, pp.165-168, the subject matter of which is incorporated herein by reference. The

removal fluid can be a compressed fluid (a category that includes supercritical fluids, near-critical fluids, expanded liquids or highly compressed gases, depending upon temperature, pressure and composition)--though fluids in their supercritical state are preferred.

Detail Description Paragraph (49):

[0057] In either case, the micro-mechanical elements are preferably first released by removing the sacrificial layer so as to free the MEMS elements (e.g. micromirrors) to move. In accordance with the invention, the organic sacrificial layer (or layers if multiple sacrificial layers are provided on the substrate) is removed with a supercritical fluid (or near-supercritical fluid). "Supercritical fluids" is the term used to describe those fluids that have been compressed beyond their critical pressure and also heated above their critical temperature. Both gases (e.g. carbon dioxide, nitrous oxide) and liquids (e.g. water) are suitable. More particularly, fluids that can be made into a supercritical fluid state for the present invention, include inorganic gases and organic gases, such as nitrogen, alkanes and preferably lower alkanes (e.g. methane, ethane, propane, butane), or alkenes, preferably lower alkenes (e.g. propylene). Also usable in the present invention are supercritical xenon, krypton, methanol, ethanol, isopropanol and isobutanol. Supercritical hydrocarbons or fluorocarbons could also be used, as well as partially fluorinated and perfluorinated halocarbons, and highly polar hydrogen bonding solvents. Other examples of supercritical fluids that could be used in the present invention include supercritical ethanol, acetic acid, xenon and ethane, and mixtures thereof.

Detail Description Paragraph (53):

[0061] The phase behavior or ternary systems of carbon dioxide and the solubilities of a large number of compounds in liquid carbon dioxide and supercritical carbon dioxide have been much studied since 1954. Carbon dioxide is not a very good solvent for high molecular weight and polar compounds (with some exceptions as noted previously). To increase the solubility of such compounds in liquid or supercritical carbon dioxide (and subcritical and near supercritical carbon dioxide), small amounts (e.g. less than 50 mol %, preferably from 0 to 25% mol %) of polar or nonpolar cosolvents can be added. These cosolvents can be used themselves as the supercritical fluid, however, more environmentally friendly substances such as water, carbon dioxide and nitrous oxide are preferred as the supercritical fluid, with the cosolvent used being a minor mol %. Cosolvents such as methane, ethane, propane, butane, etc., and methanol, ethanol, propanol, butanol, etc., as well as methylene, ethylene, propylene, butylene, etc., as well as lower hazard organic co-solvents such as methylene carbonate, ethylene carbonate, propylene carbonate, etc. as well as the chlorides of methylene, ethylene, propylene, etc. can be used. Other possible cosolvents include hexanoic acid, octanoic acid, decanoic acid, pentanoic acid, heptanoic acid, furfural, trioctylamine, isopropylamine, trioctylphosphine oxide, 2-ethyl hexanol, n-butanol, n-amy alcohol, t-amy alcohol, decyl alcohol, and mixtures thereof.

Detail Description Paragraph (54):

[0062] Many other solvents can be used for both depositing the organic sacrificial layer and removing the organic sacrificial layer (as a supercritical fluid or preferably mixed with a supercritical fluid such as carbon dioxide, water, or nitrous oxide. Examples include ethyl acetate, propionitrile, toluene, xylene, tetramethylene sulfone, cellosolve acetate. More particularly, suitable solvents which may be utilized include ketones such as acetone, methyl ethyl ketone, methyl isobutyl ketone, mesityl oxide, methyl amyl ketone, cyclohexanone and other aliphatic ketones; esters such as methyl acetate, ethyl acetate, alkyl polycarboxylic acid esters; ethers such as methyl t-butyl ether, dibutyl ether, methyl phenyl ether and other aliphatic or alkyl aromatic ethers; glycol ethers such as ethoxy ethanol, butoxy ethanol, ethoxy 2-propanol, propoxy ethanol, butoxy propanol and other glycol ethers; glycol ether esters such as butoxy ethoxy acetate, ethyl 3-ethoxy propionate and other glycol ether esters; alcohols such as methanol, ethanol, propanol, iso-propanol, butanol, iso-butanol, amyl alcohol and other aliphatic alcohols; aromatic hydrocarbons such as toluene, xylene, and other aromatics or mixtures of aromatic solvents; aliphatic hydrocarbons such as VM&P naphtha and mineral spirits, and other aliphatics or mixtures of aliphatics; nitro alkanes such as 2-nitropropane. A review of the structural relationships important to the choice of solvent or solvent blend is given by Dileep et al., Ind. Eng. Chem. (Product Research and Development) 24, p.162 (1985) and Francis, A. W., J. Phys. Chem. 58, p.1099 (1954).

Detail Description Paragraph (59):

[0067] Releasing immediately prior to the application of epoxy or other bonding is preferable (though an anti-stiction treatment or other passivation treatment (or

treatment for improving epoxy bond strength) between release and bonding may be desirable). After releasing the micromechanical structures, the remainder of the device can be treated for stiction by applying an anti-stiction layer (e.g. a self assembled monolayer). The layer is preferably formed by placing the device in a liquid or gas silane, preferably a halosilane, and most preferably a chlorosilane. Of course, many different silanes and other materials are known in the art for their ability to provide anti-stiction for MEMS structures. The anti-stiction material can be applied with standard processes, or even with a compressed fluid (e.g. supercritical nitrous oxide or carbon dioxide).

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File: PGPB

Apr 17, 2003

DOCUMENT-IDENTIFIER: US 20030073302 A1

TITLE: Methods for formation of air gap interconnects

Summary of Invention Paragraph (9):

[0008] The interhalogen or noble gas halide etchants for creating air gap dielectrics in the present invention are beneficial for their ability to not etch PECVD films and some metals in the way that an HF vapor etch harms these structures (as in the prior art). In addition, interhalogens and noble gas halides can etch silicon or silicon-containing material without leaving residue. The invention further comprises a passivation step that occurs after the etch, which could also involve a SAM (self-assembled mono-layer) material.

Detail Description Paragraph (8):

[0020] The interhalogen or noble gas halide etchants for creating air gap dielectrics in the present invention are beneficial for their ability to not etch PECVD films and some metals in the way that an HF vapor etch harms these structures (as in the prior art). In addition, interhalogens and noble gas halides can etch silicon or silicon-containing material without leaving residue. The invention further comprises a passivation step that occurs after the etch, which could also involve a SAM (self-assembled mono-layer) material.

Detail Description Paragraph (20):

[0032] The sacrificial layer comprises an organic material, a carbon compound, that is deposited by, for example, spray-on or spin-coating. In one embodiment, the organic material is mixed with a solvent and deposited on a substrate. The solvent is preferably any known solvent for dissolving the organic material to be used, such as a supercritical fluid and/or a volatile organic solvent. The solvent is selected based on good handling, spinning and film forming properties (for spin on non-supercritical embodiments). In a preferred embodiment, a supercritical fluid, such as carbon dioxide, along with a cosolvent, dissolves a polymer and deposits the dissolved polymer on a substrate as a sacrificial layer.

Detail Description Paragraph (21):

[0033] In order to release the multi-layered structure, the organic sacrificial layer (or layers if multiple sacrificial layers are provided on the substrate) is removed with a supercritical fluid (or near-supercritical fluid). "Supercritical fluids" is the term used to describe those fluids that have been compressed beyond their critical pressure and also heated above their critical temperature. Both gases (e.g. carbon dioxide, nitrous oxide) and liquids (e.g. water) are suitable. More particularly, fluids that can be made into a supercritical fluid state for the present invention, include inorganic gases and organic gases, such as nitrogen, alkanes and preferably lower alkanes (e.g. methane, ethane, propane, butane), or alkenes, preferably lower alkenes (e.g. propylene). Also usable in the present invention are supercritical xenon, krypton, methanol, ethanol, isopropanol and isobutanol. Supercritical hydrocarbons or fluorocarbons could also be used, as well as partially fluorinated and perfluorinated halocarbons, and highly polar hydrogen bonding solvents. Other examples of supercritical fluids that could be used in the present invention include supercritical ethanol, acetic acid, xenon and ethane, and mixtures thereof.

Detail Description Paragraph (24):

[0036] The phase behavior or ternary systems of carbon dioxide and the solubilities of a large number of compounds in liquid carbon dioxide and supercritical carbon dioxide have been much studied since 1954. Carbon dioxide is not a very good solvent for high molecular weight and polar compounds (with some exceptions as noted previously). To increase the solubility of such compounds in liquid or supercritical carbon dioxide

(and subcritical and near supercritical carbon dioxide), small amounts (e.g. less than 50 mol %, preferably from 0 to 25% mol %) of polar or non-polar cosolvents can be added. These cosolvents can be used themselves as the supercritical fluid, however, more environmentally friendly substances such as water, carbon dioxide and nitrous oxide are preferred as the supercritical fluid, with the cosolvent used being a minor mol %. Cosolvents such as methane, ethane, propane, butane, etc., and methanol, ethanol, propanol, butanol, etc., as well as methylene, ethylene, propylene, butylene, etc., as well as lower hazard organic co-solvents such as methylene carbonate, ethylene carbonate, propylene carbonate, etc. as well as the chlorides of methylene, ethylene, propylene, etc. can be used. Other possible cosolvents include hexanoic acid, octanoic acid, decanoic acid, pentanoic acid, heptanoic acid, furfural, trioctylamine, isopropylamine, trioctylphosphine oxide, 2-ethyl hexanol, n-butanol, n-amyl alcohol, t-amyl alcohol, decyl alcohol, and mixtures thereof.

Detail Description Paragraph (25):

[0037] Many other solvents can be used for both depositing the organic sacrificial layer and removing the organic sacrificial layer (as a supercritical fluid or preferably mixed with a supercritical fluid such as carbon dioxide, water, or nitrous oxide. Examples include ethyl acetate, propionitrile, toluene, xylene, tetramethylene sulfone, cellosolve acetate. More particularly, suitable solvents which may be utilized include ketones such as acetone, methyl ethyl ketone, methyl isobutyl ketone, mesityl oxide, methyl amyl ketone, cyclohexanone and other aliphatic ketones; esters such as methyl acetate, ethyl acetate, alkyl polycarboxylic acid esters; ethers such as methyl t-butyl ether, dibutyl ether, methyl phenyl ether and other aliphatic or alkyl aromatic ethers; glycol ethers such as ethoxy ethanol, butoxy ethanol, ethoxy 2-propanol, propoxy ethanol, butoxy propanol and other glycol ethers; glycol ether esters such as butoxy ethoxy acetate, ethyl 3-ethoxy propionate and other glycol ether esters; alcohols such as methanol, ethanol, propanol, isopropanol, butanol, iso-butanol, amyl alcohol and other aliphatic alcohols; aromatic hydrocarbons such as toluene, xylene, and other aromatics or mixtures of aromatic solvents; aliphatic hydrocarbons such as VM&P naphtha and mineral spirits, and other aliphatics or mixtures of aliphatics; nitro alkanes such as 2-nitropropane. A review of the structural relationships important to the choice of solvent or solvent blend is given by Dileep et al., Ind. Eng. Chem. (Product Research and Development) 24, p. 162 (1985) and Francis, A. W., J. Phys. Chem. 58, p. 1099 (1954).

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L4: Entry 13 of 71

File: PGPB

Aug 8, 2002

DOCUMENT-IDENTIFIER: US 20020107159 A1

TITLE: Phosphate fluorosurfactants for use in carbon dioxide

Detail Description Paragraph (32):

[0047] The invention also provides a method of the separating the surfactant from the carbon dioxide and applying the surfactant to a substrate. In particular, the method comprises providing a composition of matter comprising at least one surfactant comprising (1) at least one phosphate group and (2) a solvent comprising carbon dioxide, wherein at least a portion of the surfactant is soluble in the solvent; and applying the composition of matter onto a substrate such that the carbon dioxide separates from the surfactant and wherein the surfactant coats the substrate. Techniques for separating and applying materials to a substrate are known in the art and are described, for example, in U.S. Pat. No. 5,863,612 to DeSimone et al., the disclosure of which is incorporated herein by reference in its entirety, such as found on col. 5, line 47 through col. 6, line 11. Examples of methods for separating the surfactant include, without limitation, boiling off the carbon dioxide and leaving the surfactant behind, and precipitation of the surfactant into a non-solvent either by introducing a non-solvent to a vessel or reactor containing the surfactant or the transfer of the vessel or reactor contents to another vessel containing a non-solvent for the surfactant. In one embodiment, the separation and application steps may be carried out together and include, as an example, passing (e.g., spraying or spray-drying) a solution containing the surfactant through an orifice to form particles, powder coatings, fibers, and other coatings on the substrates. A wide variety of substrates may be employed such as, without limitation, metals, organic polymers, inorganic polymers, textiles, and composites thereof. Exemplary substrates include, without limitation, integrated circuits, silicon wafers, silicon wafers with vias containing water, low-dielectric constant surfaces used as interlayer dielectrics on integrated circuits, a MEM, a porous material, a micro-porous material, a nano-porous material, a non-woven material, surfaces to be cleaned, surfaces to be treated with passivation layers, surfaces to be coated, surfaces to be treated with a self-assembled monolayer ("SAM"), photoresist coated surfaces, optical interfaces, optical relays, optical fibers, metallized surfaces, and micromirrors. These application techniques are demonstrated for liquid and supercritical solutions. The surfactant may form a low surface energy coating on the substrate. Examples of embodiments of substrates include, without limitation, textiles, papers, fiber optics, as well as other surfaces.

Detail Description Paragraph (37):

[0052] The first composition of matter comprising water may be present in the form of a number of embodiments, such as, for example, various articles of manufacture. Examples of such embodiments include, without limitation, integrated circuits, silicon wafers, silicon wafers with vias containing water, low-dielectric constant surfaces used as interlayer dielectrics on integrated circuits, a MEM, a porous material, a micro-porous material, a nano-porous material, a non-woven material, surfaces to be cleaned, surfaces to be treated with passivation layers, surfaces to be coated, surfaces to be treated with a self-assembled monolayer ("SAM"), photoresist coated surfaces, optical interfaces, optical relays, optical fibers, metallized surfaces, and micromirrors.

CLAIMS:

2. The method according to claim 1, wherein the first composition of matter is present in an embodiment selected from the group consisting of integrated circuits, silicon wafers, silicon wafers with vias containing water, low-dielectric constant surfaces used as interlayer dielectrics on integrated circuits, a MEM, a porous material, a micro-porous material, a nano-porous material, a non-woven material, surfaces to be

cleaned, surfaces to be treated with passivation layers, surfaces to be coated, surfaces to be treated with a self-assembled monolayer ("SAM"), photoresist coated surfaces, optical interfaces, optical relays, optical fibers, metallized surfaces, and micromirrors.

24. The method according to claim 23, wherein the substrate is selected from the group consisting of integrated circuits, silicon wafers, silicon wafers with vias containing water, low-dielectric constant surfaces used as interlayer dielectrics on integrated circuits, a MEM, a porous material, a micro-porous material, a nano-porous material, a non-woven material, surfaces to be cleaned, surfaces to be treated with passivation layers, surfaces to be coated, surfaces to be treated with a self-assembled monolayer, photoresist coated surfaces, optical interfaces, optical relays, optical fibers, metallized surfaces, and micromirrors.

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File: PGPB

Aug 29, 2002

DOCUMENT-IDENTIFIER: US 20020119398 A1

TITLE: CO₂-processes photoresists, polymers, and photoactive compounds for microlithographySummary of Invention Paragraph (15):

[0012] The step of contacting the substrate which results in the formation of a coating thereon may be carried out using various techniques. Examples of embodiments include, without limitation, a spin coating method, a dip coating method, and a spray coating method. A spin coating method is preferred, and is described in U.S. Pat. No. 6,001,418 to DeSimone et al., the disclosure of which is incorporated herein by reference in its entirety. A meniscus coating technique can also be employed such as one described, for example, in U.S. Pat. No. 6,083,565 to Carbonell et al., the disclosure of which is incorporated herein by reference in its entirety. A coating method using self-assembling monolayers (SAMs) can also be employed, and may be used in conjunction with CO.sub.2. Examples of molecules that are capable of producing SAMs include, without limitation, alkane thiols, fluorinated alkane thiols, and functionalized alkane thiols such as those represented by the formula:

Summary of Invention Paragraph (18):

[0014] A wide range of components can be used in the present invention to form a coating on the substrate. For the purposes of the invention, the term "materials" can include, without limitation, precursors, monomers, polymeric materials, and the like. Polymers, polymeric precursors, and monomers that are soluble or insoluble in CO.sub.2 or CO.sub.2 mixed with other gases (insert or otherwise) and co-solvents including surfactants may be necessary to maintain low surface tension and solubility and viscosity as CO.sub.2 evaporates, at a number of density and/or pressure conditions are encompassed by the invention. Examples of suitable embodiments include polymeric materials which comprise, without limitation, fluoropolymers, silicon-containing polymers, or combinations thereof, such polymers having a CO.sub.2-soluble or "CO.sub.2-philic" component. "CO.sub.2-philic" refers to the polymer having an affinity for carbon dioxide. Examples of suitable monomers for making the fluoropolymers are numerous and include, but are not limited to, those set forth in U.S. Pat. No. 5,739,223 to DeSimone et al., the disclosure of which is incorporated herein by reference in its entirety. Carbon dioxide soluble monomers may be employed if so desired. Exemplary monomers are fluoroacrylate monomers, fluorostyrene monomers, fluoroalkylene oxide monomers, fluoroolefin monomers, fluorinated alkyl vinyl ether monomers, cyclic fluorinated monomers, and mixtures thereof. Specific preferred monomers include, without limitation, 2-(N-ethylperfluorooctane-sulfonamido) ethyl acrylate, 2-(N-ethylperfluorooctane-sulfonamido) ethyl methacrylate, 2-(N-methylperfluorooctane-sulfonamido) ethyl acrylate, 2-(N-methylperfluorooctane-sulfonamido) ethyl methacrylate, 1,1'-dihydroperfluorooctyl acrylate, 1,1'-dihydroperfluorooctyl methacrylate, 1,1',2,2'-tetrahydroperfluoroalkylacrylate, 1,1',2,2'-tetrahydroperfluoroalkyl methacrylate, .alpha.-fluorostyrene, 2,4,6-trifluoromethylstyrene, hexafluoropropylene oxide, perfluorocyclohexane oxide, tetrafluoroethylene, vinylidene fluoride, chlorotrifluoroethylene, perfluoro(propyl vinyl ether), perfluoro(methyl vinyl ether), 2,2-bis-trifluoromethyl-4,5-difluoro-1,3-dioxole, and mixtures thereof, including those set forth in U.S. Pat. No. 6,083,565 to Carbonell et al., the disclosure of which is incorporated herein in its entirety by reference.

Summary of Invention Paragraph (35):

[0031] In one embodiment, and in accordance with the invention, one is capable of designing the resist such that it can be deposited by utilizing conventional techniques, i.e., known solvents, then developed by employing a first fluid or composition comprising carbon dioxide at a given density, and then stripping the resist

using a second fluid or composition comprising carbon dioxide, either in a liquid or supercritical state. The first and second fluids or compositions may be the same or different. Thus, it is believed that the invention is distinguished from U.S. Pat. No. 5,665,527 to Allen et al.

Detail Description Paragraph (61):

[0128] This invention addresses problems associated with image collapse, resist transparency at 157 nm, potential solubility problems of new resist materials in a readily available solvent, elimination of organic solvents and aqueous developers (i.e., turning a wet process into a dry process). The invention uses differences in polarity to create solubility differences in supercritical and liquid CO.sub.2.

CLAIMS:

16. The process according to claim 2, wherein said step of contacting the substrate with a first composition comprising carbon dioxide and a component comprises coating the component using a method selected from the group consisting of a spin coating method, a dip coating method, a meniscus coating method, a coating method using self-assembled monolayers, and a spray coating method.

32. The process according to claim 18, wherein said step of contacting the substrate with a first composition comprising carbon dioxide and a component comprises coating the component using a method selected from the group consisting of a spin coating method, a dip coating method, a meniscus coating method, a coating method using self-assembled monolayers, and a spray coating method.

51. The process according to claim 50, wherein said step of contacting the substrate with a first composition comprising carbon dioxide and a component comprises coating the component using a method selected from the group consisting of a spin coating method, a dip coating method, a meniscus coating method, a coating method using self-assembled monolayers, and a spray coating method.

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L4: Entry 28 of 71

File: USPT

Feb 6, 2001

DOCUMENT-IDENTIFIER: US 6183815 B1

TITLE: Method and composition for surface treatment of metals

Brief Summary Text (24):

In alternative embodiment, liquid carbon dioxide can be used as the solvent. In this case, the coating process would have to be carried out under pressure, so as to avoid rapid evaporation of the CO.sub.2 solvent. On the other hand, use of liquid CO.sub.2 as a solvent has the advantage of allowing rapid drying of the surface (by depressurization) once the desired self assembly period has elapsed. Liquid CO.sub.2 has the further advantage of being non-toxic, non-flammable, and environmentally safe.

Brief Summary Text (28):

Once the surface is ready for coating the following steps are preferably carried out. The solution is sprayed onto the surface using a pump sprayer with a mist discharge until the entire metal surface is wet. In order to minimize runoff, it is preferred to place the object in a position such that its surfaces are as horizontal as possible. The surface is left undisturbed for a time sufficient to allow the solvent in the film on the surface to evaporate. The concentration of thiol in the solvent must be high enough for a self-assembled monolayer to form before the solvent evaporates. Depending on the solvent employed, the thiol molecules will require 0.1-10 minutes of wet time to self assemble a coating layer on the metal surface. Therefore, it is preferred that the solvent not evaporate in less than 0.1 minute.

Brief Summary Text (29):

Once the self assembled layer has had time to form, it is necessary to remove the excess thiol from the surface. The thin film of liquid sprayed on the surface of the metal contains a great excess of thiol. This high concentration is required to drive the self-assembly of the thiol before the solvent evaporates. The excess thiol deposits in a non-oriented, non-aligned manner on top of the monolayer. It is undesirable to leave excess thiol on top of the self-assembled monolayer, as its unassembled nature causes the surface to be less hydrophobic and its presence can be visible to the naked eye in the form of "rainbows" of color on the surface or brown deposits of very thick layers. The removal of excess thiol can be accomplished by either polishing the metal with a soft cloth and/or by rinsing the metal surface thoroughly with water for 10-30 seconds. When the excess thiol is removed, the water will start to "bead up" on the coated surface, indicating that only the hydrophobic self-assembled monolayer remains on the metal.

Detailed Description Text (2):

A solution comprising 1.0 wt. % of F(CF₂)₆CONH(CH₂)₂SH (tridecafluorohexyl amide ethylthiol) in 99.0 wt. % aqueous isopropyl alcohol is sprayed onto a silver surface. The thiol has no detectable odor before or after dissolution in the alcohol. The alcohol comprises 91 vol. % isopropyl alcohol and 9 vol. % water. The surface dries in several minutes, and is then rinsed and dried with a soft cloth. The surface is coated with a monolayer that is hydrophobic, is not detectable to the naked eye, and is not readily removed by rubbing. When exposed to a "tarnish chamber" that provides a very corrosive environment (high SO.sub.2 concentration) for silver, the coated surface did not tarnish, while a similar metal surface that was not coated tarnished severely. The coating does not discolor, crack or peel. Surface analysis indicates that a uniform, self-assembled monolayer has been established on the silver.

Detailed Description Text (4):

A solution comprising 1.0 wt. % of F(CF₂)₆CONH(CH₂)₂SH (tridecafluorohexyl amide ethylthiol) in 99.0 wt. % isopropyl alcohol is sprayed onto a silver surface. The thiol has no detectable odor before or after dissolution in the alcohol. The alcohol

comprises 100 vol. % isopropyl alcohol. The surface dries in several minutes, and is then rinsed and dried with a soft cloth. The surface is coated with a monolayer that is hydrophobic, is not detectable to the naked eye, and is not readily removed by rubbing. When exposed to a high SO₂ concentration, the coated surface did not tarnish, while a similar metal surface that was not coated tarnished severely. The coating does not discolor, crack or peel. Surface analysis indicates that a uniform, self-assembled monolayer has been established on the silver.

Detailed Description Text (6):

A solution comprising 1.0 wt. % of F(CF₂)₆CONH(CH₂)₂SH (tridecafluorohexyl amide ethylthiol) in 99.0 wt. % normal propanol is sprayed onto a silver surface. The thiol has no detectable odor before or after dissolution in the alcohol. The alcohol comprises 100 vol. % normal propanol. The surface dries in several minutes, and is then rinsed and dried with a soft cloth. The surface is coated with a monolayer that is hydrophobic, is not detectable to the naked eye, and is not readily removed by rubbing. When exposed to a high SO₂ concentration, the coated surface did not tarnish, while a similar metal surface that was not coated tarnished severely. The coating does not discolor, crack or peel. Surface analysis indicates that a uniform, self-assembled monolayer has been established on the silver.

Other Reference Publication (1):

Schönherr et al., Lattice Orientation and Tribology of SAMs of Fluorinated Thiols and Disulfides on Au(111) by AFM: The Influence of the Molecular Structure, Polymer Preprints, pp. 904-905, Aug. 1998.

Other Reference Publication (2):

Schönherr et al., Self-Assembled Monolayers of Symmetrical and Mixed Alkyl Fluoroalkyl Disulfides on Gold. 1. Synthesis of Disulfides and Investigation of Monolayer Properties, Langmuir, pp. 3891-3897, Feb. 1996.

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REPRESENTATIVE-FIGURES: 1

ABSTRACT:

A self-assembled monolayer (SAM) is fabricated using either a semi-fluorinated sulphur containing compound, or a semi-fluorinated silane derivative and compressed carbon dioxide (CO.sub.2) as the solvent medium. The temperature and/or pressure of the compressed CO.sub.2 may be varied during the fabrication process to improve the molecular packing density of the monolayer.

By using compressed CO.sub.2 as the solvent medium, monolayers with good molecular packing density can be fabricated relatively quickly without the use of environmentally unfriendly solvents.

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Luscombe, Christine K.	Cambridge		GB	

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CLAIMS:

1) A method of fabricating a self-assembled monolayer of a substance on a substrate comprising depositing the substance on the substrate using compressed carbon dioxide as the solvent medium for the substance.

2) A method as claimed in claim 1, wherein the pressure and/or temperature of the compressed carbon dioxide is/are selectively controlled so as to enhance the density of the self-assembled monolayer on the substrate.

3) A method as claimed in claim 1 comprising the use of a co-solvent in combination with the compressed carbon dioxide.

4) A method as claimed in claim 3, wherein the co-solvent comprises at least one of H.sub.2O, CH.sub.3OH, CF.sub.3OH, CF.sub.3CH.sub.2OH, CF.sub.3CF.sub.2OH, (CF.sub.3).sub.2CHOH, CH.sub.4, C.sub.2H.sub.4, C.sub.2F.sub.6, CHF.sub.3, CClF.sub.3, C.sub.2H.sub.6, SF.sub.6, Propylene, Propane, NH.sub.3, Pentane, .sup.1PrOH, MeOH, EtOH, .sup.1BuOH, Benzene, Pyridine.

5) A method as claimed in claim 1, wherein the substrate comprises a metallic substance.

6) A method as claimed in claim 5, wherein the metallic substance comprises at least one of gold, silver, copper, iron, mercury, palladium, gallium arsenide, ferrous oxide, indium tin oxide.

7) A method as claimed in claim 6, wherein the substance comprises a semi-fluorinated sulphur containing compound of the formula: 3Where X comprises R--SH, RS--SR or R--S--R, where R denotes the rest of the molecule; Y comprises a functional group; and m and n denote respectively the number of fluorinated and non-fluorinated carbon atoms.

8) A method as claimed in claim 7, wherein X comprises a disulphide of sulphur.

9) A method as claimed in claim 7, wherein X comprises a thiol.

- 10) A method as claimed in claim 7, wherein Y comprises a CF.sub.3 functional group.
- 11) A method as claimed in claim 7, wherein m and n lie within the range of 1 to 20.
- 12) A method as claimed in claim 11, wherein m and n lie within the range of 5 to 10.
- 13) A method as claimed in claim 12, where m is 8 and n is 10.
- 14) A method as claimed in claim 7, wherein Y further comprises at least one of vinyl, styryl, acryloyl, methacryloyl or alkyne in combination with a spacer group.
- 15) A method as claimed in claim 14, wherein the spacer group comprises at least one of CH.sub.2 or CF.sub.2.
- 16) A method as claimed in claim 1, wherein the substrate comprises at least one of glass, mica, SiO.sub.2, Al.sub.2O.sub.3, Ga.sub.2O.sub.3 or ITO.
- 17) A method as claimed in claim 16, wherein the substance comprises a semi-fluorinated silane derivative of the formula: 4where Y comprises a functional group; and m and n denote respectively the number of fluorinated and non-fluorinated carbon atoms.
- 18) A method as claimed in 17, wherein Si comprises a trialkoxy derivative.
- 19) A method as claimed in claim 18, wherein Si comprises at least one of SiCl.sub.3, Si(OCH.sub.3).sub.3, Si(OCH.sub.2CH.sub.3).sub.3, Si(OCH.sub.3).sub.2Cl or Si(CH.sub.2CH.sub.3).sub.2Cl.
- 20) A method as claimed in claim 17, wherein Y comprises a CF.sub.3 functional group.
- 21) A method as claimed in claim 17, wherein m and n lie within the range of 1 to 20.
- 22) A method as claimed in claim 21, wherein m and n lie within the range of 5 to 10.
- 23) A method as claimed in claim 22, wherein m is 6 and n is 1.
- 24) A method as claimed in claim 17, wherein Y further comprises at least one of vinyl, styryl, acryloyl, methacryloyl or alkyne in combination with a spacer group.
- 25) A method as claimed in claim 24, wherein the spacer group comprises at least one of CH.sub.2 or CF.sub.2.
- 26) A method as claimed in claim 1, wherein the self-assembled monolayer has an ellipsometry thickness of about 30 .ANG. and a water contact angle of about 110.degree..
- 27) An inkjet head comprising a self-assembled monolayer as claimed in any one of claims 1 to 15 or claim 26, when appendant to any one of claims 1 to 15.
- 28) An electronic, optical or optoelectronic device comprising a self-assembled monolayer as claimed in any one of claims 1 to 5 or claims 16 to 26 or claim 26 when appendant to any one of claims 1 to 5, or 16 to 25.
- 29) A device as claimed in claim 28 comprising a thin film transistor or an organic semiconductor device, or a light emitting diode.
- 30) A device as claimed in claim 29, wherein the light emitting diode comprises an organic polymer light emitting diode.